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TECHNICAL NOTE

No. 1608

QUANTITATIVE ANALYSIS OF TERNARY MIXTURES OF NAPHTHALENE

1 -METHYLNAPHTHALENE, AND 2-METHYLNAPHTHALENE

BY ULTRAVIOLET SPECTROPHOTOMETRY

By Alden P. Cleaves, Mildred S. Carver, and Robert R. Hibbard

Flight Propulsion Research Laboratory
Cleveland, Ohio



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SUMMARY

A method for quantitative analysis of ternary mixtures of naphthalene, 1-methylnaphthalene, and 2-methylnaphthalene on the basis of ultraviolet absorption in the region from 3000 to 3300 Å is presented and the effect of interference by a fourth absorber was investigated. Some data pertinent to possible interference by sulfur compounds of the types expected in petroleum are also included.

Twelve synthetic ternary mixtures of the three naphthalenes were analyzed with an average error of 0.8 percent of the alkyl-naphthalenes present.

The method was applied to seven cuts of American crude petroleum that distilled at temperatures, which indicated that the three lowest boiling naphthalenes constituted nearly all the condensed dicyclic aromatics present. Accuracy in the case of the cuts of crudes could not be definitely established but was estimated to be about 0.03 percent of the sample.

INTRODUCTION

The steadily increasing use of high-boiling hydrocarbon fuels (that is, those distilling above the gasoline range) in jet-propulsion and Diesel engines has led to recent extensive research to determine whether the efficiency and the performance of these engines may be markedly affected by some specific compound or by a particular type of hydrocarbon. These trends have made analysis of such hydrocarbon mixtures increasingly important.

Applications of analytical methods established for gasoline to the higher boiling range of kerosene and Diesel oil often change

the significance of these methods or render them inadequate. Condensed dicyclic aromatic hydrocarbons constitute one type that is present in kerosene but not in gasoline. Chemical and physical methods for determining amounts of this type of compound are described in references 1 to 3. The polarographic method of reference 4 determines total naphthalenes in petroleum fractions for cases in which naphthalenes are the only polynuclear aromatics present. These methods could be specific for a particular naphthalene only if it was isolated by fractionation.

The possibility of using ultraviolet spectrophotometry for the identification and estimation of alkylnaphthalenes in straight-run petroleum distillates was proposed in reference 5. No method of analysis for the individual compounds, 1-methylnaphthalene and 2-methylnaphthalene, was established, however, in reference 5. Instead a method of estimation was proposed on the basis of an isomeric mixture of these compounds adopted as typical.

A method developed at the NACA Cleveland laboratory and described herein supplements reference 5 by demonstrating the possibility of quantitative analysis of mixtures of the three alkylnaphthalenes that have lowest boiling points. Paraffins, naphthenes, olefins, monocyclic and noncondensed polycyclic aromatics do not interfere with the method. The possibility of interference by some sulfur compounds that are present in petroleum crudes of high sulfur content was considered and the ultraviolet spectra of four compounds representing three types of sulfur compound are included. The usual procedure of analyzing synthetic ternary mixtures of the purest compounds available and comparing the concentrations determined with the known compositions has been used to establish the accuracy of the method. As an analogy of the case in which distillation might not perfectly isolate the three alkylnaphthalenes, the effect of interference by a known amount of a fourth absorber has been investigated. Seven cuts of American crudes have also been analyzed and the accuracy of determining a known amount of naphthalene or a 2-methylnaphthalene added to one of the cuts was evaluated.

INSTRUMENTS AND MATERIALS

The quartz photoelectric spectrophotometer used for the measurements is a commercially available instrument described in reference 6. The instrument was modified to give greater sensitivity (and thus allow the use of narrower slits) by substituting a 5000-megohm resistor for the 2000-megohm resistor designated R_{10} in the electrical circuit diagram (fig. 5) of reference 6. A hydrogen discharge lamp was the source of ultraviolet light. Extinctions were read directly from a calibrated dial. The widths

of entrance and exit slits were adjusted by a single knob and were set at either 0.16 or 0.50 millimeter. The isooctane solvent and the solutions of the samples in isooctane were held in matched quartz cells 0.50 centimeter thick. Small quantities of sample were weighed on an analytical balance.

The dicyclic aromatics in the following list were used in the investigation:

Hydrocarbon	Boiling point		Analytical wavelength (A)
	(°F)	(°C)	
Naphthalene	424	218	3110
2-Methylnaphthalene	466	241	3190
1-Methylnaphthalene	473	245	3140

The naphthalene was recrystallized from ethanol until its melting point was 80° C (176° F). The 1-methylnaphthalene, synthesized and purified at Western Reserve University, Cleveland, Ohio, was furnished by Dr. Oliver J. Grummitt. The 2-methylnaphthalene was an Eastman Kodak Company "white label" chemical recrystallized twice from ethanol to improve its purity and homogeneity. These chemicals were probably above 98 percent in purity. The acenaphthene was used as supplied under "white label" by Eastman Kodak Company.

The phenyl sulfide, amyl sulfide, dodecyl sulfide, and dodecyl mercaptan were used as supplied by manufacturers and were probably above 90 percent in purity.

PROCEDURE AND RESULTS

The ultraviolet absorption spectra of naphthalene, 1-methylnaphthalene, and 2-methylnaphthalene were measured in the range from 3010 to 3240 A with slits set at 0.16 millimeter (fig. 1). (See reference 5 for the naphthalene spectrum measured with 0.50-mm slits.) Measurements at several of the peaks of absorption were also made with slits 0.50 millimeter wide. The narrower slit width is near the minimum that could be used with the modified instrument and the wider slit width is in the middle of the range available to users of the unmodified spectrometer. Calibrations were made and test analyses run at both these slit widths to demonstrate the accuracy of the proposed method under

both improved and normal spectrometer-sensitivity conditions. The spectrum of acenaphthene was measured in the region from 3085 to 3295 Å with 0.50-millimeter slits (fig. 2). The spectra of the sulfur compounds were measured from 2200 to 3300 Å with 0.50-millimeter slits (fig. 3).

In the 12 ternary mixtures prepared, three were blends of approximately equal concentrations of each of the three alkylnaphthalenes. The remaining nine mixtures were divided into three groups; one of the components of each group was in low concentration relative to the other two. Two synthetic quaternary mixtures contained these three alkylnaphthalenes in approximately equal amounts and about 1 percent by weight of acenaphthene based on total alkylnaphthalenes to serve as an interferer.

The mixtures were so diluted with isooctane that the total alkylnaphthalene concentration was approximately 0.50 gram per liter; the absorption in a 0.50-centimeter cell was measured at the wavelengths chosen for analysis at slit widths of 0.16 and 0.50 millimeter. Solution of three simultaneous linear equations as in reference 7 yielded the spectrophotometric analyses.

The following example of an application of these simultaneous equations is given for the 0.16-millimeter slit:

$$E_{3110} = 1.942c_1 + 1.784c_2 + 2.062c_3$$

$$E_{3140} = 0.516c_1 + 2.341c_2 + 1.541c_3$$

$$E_{3190} = 0.137c_1 + 0.797c_2 + 3.718c_3$$

where E_{3110} , E_{3140} , and E_{3190} are the extinctions at the three analytical wavelengths as indicated by the spectrometer and c_1 , c_2 , and c_3 are the concentrations in grams per liter of naphthalene, 1-methylnaphthalene, and 2-methylnaphthalene, respectively. The numerical coefficients are values of the specific extinctions k , which are the absorptions by solutions of 1 gram per liter in a 1-centimeter cell of the respective compounds at the designated wavelengths. The calculations are based on a 1-centimeter cell and the extinctions are twice the value of the measured optical density.

For test solution 1, substitution of the experimentally determined values of 0.989, 0.667, and 0.733 for E_{3110} , E_{3140} , and E_{3190} and simultaneous solution of the three equations yields

concentrations of 41.3, 24.8, and 30.5 percent by weight for c_1 , c_2 , and c_3 , respectively, as shown in the first row of table I.

Results of the analyses of ternary mixtures of the alkyl-naphthalenes with 0.16-millimeter slits are given in table I and results of similar analyses with 0.50-millimeter slits are given in table II. Differences between the known concentrations calculated from the weights of compounds in the mixtures and the concentrations determined by spectrophotometric analysis are also tabulated as percentages of total alkyl-naphthalenes and averaged in tables I and II. In table III are shown the results obtained when small amounts of acenaphthene (about 1 percent based on total alkyl-naphthalenes) were added to two synthetic mixtures. Concentrations of the three principal constituents in these two mixtures were calculated as if acenaphthene were not present in order to demonstrate effects of an unsuspected interferer. The true synthetic concentrations are also given.

The analyses of six fractions (A.S.T.M. end points below 420° F (215.6° C)) from Tomball, Bradford, Midway, Yates, and Hastings crudes are given in table IV together with A.S.T.M. distillation data. These low-boiling fractions were used because the end points made it unlikely that interfering higher-boiling condensed polynuclear hydrocarbons would be present in significant quantities. In table V are shown the results obtained when known amounts of naphthalene and 2-methylnaphthalene were added to the higher-boiling cut of Hastings crude (278° to 420° F). The ultraviolet spectra showing these additions are given in figure 4. These known additions were determined to within 0.03 percent of sample; hence, it is estimated that the accuracy of the results in table IV is about 0.03 percent of the total sample.

DISCUSSION OF RESULTS

The method proposed herein is applicable to concentrations ranging from 0.002 to 100.0 percent by weight. The average of differences between known and analyzed concentrations for the 12 ternary mixtures was 0.8 percent by weight of the total alkyl-naphthalenes for measurements with 0.16-millimeter slits and 1.2 percent for measurements with 0.50-millimeter slits. The lower accuracy at the wider slit setting was expected and the 0.8-percent deviation was considered satisfactorily low. The result that deviations in the determinations of low concentrations were in nearly all cases less than the average when 0.16-millimeter slits were used was unexpected. Inspection of tables I and II

shows no significant trend in deviations with respect to sign. Differences between the average deviations for each component are hardly large enough to be significant but they were slightly lower in the case of 2-methylnaphthalene.

Isooctane, a transparent paraffinic solvent, constituted more than 99 percent of each of the solutions measured in the spectrophotometer. Naphthenes, olefins, conjugated olefins, monocyclic, and noncondensed polycyclic aromatics are also essentially transparent in the 3100 to 3300 Å region; hence, the analyses may be performed in the presence of these types of hydrocarbon compound. The problem of obtaining samples that contain only naphthalene and the methylnaphthalenes from crudes that may contain numerous compounds in the alkylnaphthalene series has not been investigated. Ethylnaphthalenes and propylnaphthalenes are the most probable interferers but because of their unavailability at this time the tests with acenaphthene (b.p., about 538° F (281° C)) were substituted. The boiling point of acenaphthene is above the range for the alkylnaphthalenes considered but it is one of the most serious possible interferers known and has absorption that averages about three times as intense at the analytical wavelengths as the absorption by each of the other components. The presence of approximately 1 percent by weight of acenaphthene (based on total alkylnaphthalenes) purposely added to mixtures 13 and 14, but treated as an unsuspected interferer, yielded an average deviation of 1.2 percent for measurements with 0.16-millimeter slits. This deviation is indeed small. The value of 4.1 percent obtained when slits were 0.50 millimeter wide is nearer the expected value.

In general, the spectra of the sulfur compounds shown in figure 3 indicate that they would cause slightly more serious interference with determinations of monocyclic aromatics as proposed in reference 7 than with the present method for alkylnaphthalenes. Consequently, the monocyclic aromatic absorption region from 2200 to 2800 Å is also included in these spectra. On the basis of these sulfur-compound spectra, the presence of 1 percent of these compounds on the basis of total alkylnaphthalenes would be expected to cause the sum of alkylnaphthalene determinations to be high by not more than the following percentages:

Sulfur compounds	Increase due to presence of 1 percent of compound (percent)
Dodecyl mercaptan	0.0007
Dodecyl sulfide	.03
Amyl sulfide	.04
Phenyl sulfide	.3

The boiling point of phenyl sulfide is about 556°F (296°C), hence it would probably be reduced to a negligible concentration if ethylnaphthalenes were successfully removed from a sample by distillation. In the case of some cuts from crude petroleum of high sulfur content, selective removal of the sulfur compounds would probably be necessary.

SUMMARY OF RESULTS

On the basis of ultraviolet spectrophotometric measurements in the region from 3000 to 3300 Å, the following results have been obtained:

Synthetic ternary mixtures of naphthalene, 1-methylnaphthalene, and 2-methylnaphthalene were quantitatively analyzed with an average error of 0.8 percent of the alkylnaphthalenes present.

The method was applied to cuts of crude petroleum and the average error was estimated to be about 0.03 percent of the total sample.

Flight Propulsion Research Laboratory,
National Advisory Committee for Aeronautics,
Cleveland, Ohio, November 12, 1947.

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TABLE I - ANALYTICAL RESULTS ON SYNTHETIC MIXTURES OF ALKYLNAPHTHALENES
USING ULTRAVIOLET SPECTROPHOTOMETER WITH 0.16-MILLIMETER SLITS

Solution	Total alkyl-naphthalenes in solution (gram/liter)	Known composition (percent by weight)			Compositions by analysis and differences between analyzed and known compositions (percent by weight)						Average percentage difference per solution
		Naphthalene	1-Methyl-naphthalene	2-Methyl-naphthalene	Naphthalene		1-Methylnaphthalene		2-Methylnaphthalene		
					Anal- yzed	Differ- ence	Anal- yzed	Differ- ence	Anal- yzed	Differ- ence	
1	0.5279	43.6	25.0	31.5	41.3	-2.3	24.8	-0.2	30.5	-1.0	1.2
2	.6875	30.1	28.3	41.6	30.6	.5	26.7	-1.6	41.3	-.3	.8
3	.5391	28.8	31.4	39.8	27.2	-1.6	30.5	-.9	37.2	-2.6	1.7
4	.4570	7.5	41.1	51.3	7.0	-.5	41.7	.6	50.5	-.8	.6
5	.6198	8.2	40.8	51.0	6.8	-1.4	37.8	-3.0	51.9	.9	1.8
6	.5290	4.3	53.1	42.6	3.8	-.5	53.5	.2	43.3	.7	.5
7	.5839	56.2	3.2	40.5	57.4	1.2	2.7	-.5	40.8	.1	.6
8	.5184	52.6	6.2	41.2	53.2	.6	6.5	.1	40.6	-.6	.4
9	.7093	48.1	5.3	45.6	48.6	-.5	5.2	-.1	44.4	-1.2	.6
10	.5442	60.7	35.2	4.1	60.3	-.4	37.0	1.8	4.1	0	.7
11	.3717	46.9	45.2	7.9	46.8	-.1	46.8	1.6	8.2	.3	.7
12	.7808	34.8	63.1	2.1	34.7	-.1	63.2	.1	1.7	-.4	.2
Average difference per component						0.8		0.9		0.7	
Over-all average difference						0.6					

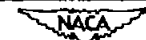


TABLE II - ANALYTICAL RESULTS ON SYNTHETIC MIXTURES OF ALKYLNAPHTHALENES USING 0.50-MILLIMETER SLITS

Solution	Total alkyl-naphthalenes in solution (gram/liter)	Known composition (percent by weight)			Composition by analysis and differences between analyzed and known compositions (percent by weight)						Average percentage difference per solution
		Naphthalene	1-Methyl-naphthalene	2-Methyl-naphthalene	Naphthalene		1-Methylnaphthalene		2-Methylnaphthalene		
					Analyzed	Difference	Analyzed	Difference	Analyzed	Difference	
1	0.5279	43.6	25.0	31.5	44.0	0.4	24.1	-0.9	32.5	1.0	0.8
2	.6875	30.1	28.3	41.6	31.5	1.4	26.7	-1.6	41.3	-.3	1.1
3	.5391	28.8	31.4	39.8	25.3	-3.5	30.8	-.6	38.4	-1.4	1.8
4	.4570	7.5	41.1	51.3	6.1	-1.4	41.9	.8	52.0	.7	1.0
5	.6198	8.2	40.8	51.0	9.8	1.8	37.5	-3.3	50.1	-.9	1.9
6	.5290	4.3	53.1	42.6	2.6	-1.7	53.7	.6	41.9	-.7	1.0
7	.5839	56.2	3.2	40.5	57.6	1.4	.7	-2.5	42.1	1.6	1.8
8	.5184	52.6	6.2	41.2	51.4	-1.2	5.5	-.7	42.3	1.1	1.0
9	.7093	49.1	5.3	45.6	48.5	-.6	4.4	-.9	44.4	-1.2	.9
10	.5442	60.7	35.2	4.1	61.3	.6	36.9	1.7	4.1	0	.8
11	.3717	46.9	45.2	7.9	47.7	.8	47.2	2.0	8.3	.4	1.1
12	.7806	34.8	63.1	2.1	34.3	-.5	63.1	0	1.0	-1.1	.5
Average difference per component						1.3		1.3		0.9	
Over-all average difference						1.2					



TABLE III - EFFECT OF SMALL AMOUNTS OF ACENAPHTHENE ON ACCURACY OF ALKYLNAPHTHALENE DETERMINATION
USING ULTRAVIOLET SPECTROPHOTOMETER WITH 0.16- AND 0.50-MILLIMETER SLITS

Solution	Total alkyl-naphthalenes in solution (gram/liter)	Known composition based on alkyl-naphthalenes (percent by weight)			Acenaphthene added in percent of alkyl-naphthalenes	Compositions by analysis and differences between analyzed and known compositions (percent by weight)						Average percentage difference per solution	
		Naphthalene	1-Methyl-naphthalene	2-Methyl-naphthalene		Naphthalene		1-Methyl-naphthalene		2-Methyl-naphthalene			
						Anal- yzed	Differ- ence	Anal- yzed	Differ- ence	Anal- yzed	Differ- ence		
With 0.16-millimeter slits													
13	0.5745	28.8	31.4	39.8	1.4	31.3	2.5	32.6	1.2	40.0	0.2	1.3	
14	.8522	49.2	5.3	45.5	1.0	51.7	2.5	5.8	.5	45.3	-.2	1.1	
Average difference per component							2.5		0.9		0.2		
Over-all average difference							1.2						
With 0.50-millimeter slits													
13	0.5745	28.8	31.4	39.8	1.4	30.7	1.9	31.4	0	41.6	1.8	1.2	
14	.8522	49.2	5.3	45.5	1.0	53.8	14.6	-.2	-5.5	46.4	.9	7.0	
Average difference per component							8.3		2.8		1.4		
Over-all average difference							4.2						



TABLE IV - ANALYTICAL RESULTS ON STRAIGHT RUN FRACTIONS OF AMERICAN CRUDES

Crude	Percent evaporated				Volume alkyl-naphthalenes found (percent)							
	10	50	90	End point	0.16-millimeter slits				0.50-millimeter slits			
	A.S.T.M. distillation temperature (°F)				Naphtha- lene	1-Methyl- naphtha- lene	2-Methyl- naphtha- lene	Total alkyl- naphtha- lenes	Naphtha- lene	1-Methyl- naphtha- lene	2-Methyl- naphtha- lene	Total alkyl- naphtha- lenes
Tomball 1A	224	262	315	338	0.014	0.012	0.005	0.031	0.014	0.012	0.007	0.033
Bradford 2A	138	234	311	372	.021	.038	.022	.081	.015	.039	.029	.083
Midway 3A	159	262	357	396	.129	.072	.059	.239	.124	.072	.069	.265
Yates 4A	203	259	311	356	.064	.067	.035	.166	.061	.042	.062	.175
Hastings 5A	158	215	272	319	.006	.005	.002	.013	.006	.005	.003	.014
Hastings 5B	308	343	388	420	.304	.070	.102	.476	.289	.062	.109	.460



TABLE V - TWO CASES OF RECOVERY OF AN ALKYLNAPHTHALENE ADDED TO A CUT OF HASTINGS CRUDE

Sample	Alkyl-naphthalene, percent by volume							
	With 0.16-millimeter slits				With 0.50-millimeter slits			
	Naphtha- lene	1-Methyl- naphtha- lene	2-Methyl- naphtha- lene	Total alkyl- naphtha- lene	Naphtha- lene	1-Methyl- naphtha- lene	2-Methyl- naphtha- lene	Total alkyl- naphtha- lene
Hastings 5B, initial composition ^a	0.304	0.070	0.102	0.476	0.289	0.062	0.109	0.460
Added alkyl-naphthalene	.440	.000	.000	.440	.440	.000	.000	.440
Calculated composition	.744	.070	.102	.916	.729	.062	.109	.900
Analyzed composition	.724	.066	.115	.905	.757	.049	.115	.921
Differences	-.020	-.004	.013	-.011	.028	-.013	.006	.021
Hastings 5B, initial composition ^a	0.304	-0.070	0.102	0.476	0.289	0.062	0.109	0.460
Added alkyl-naphthalene	.000	.000	.454	.454	.000	.000	.454	.454
Calculated composition	.304	.070	.556	.930	.289	.062	.563	.914
Analyzed composition	.296	.047	.546	.889	.328	.047	.530	.905
Differences	-.008	-.023	-.010	-.041	.039	-.015	-.033	-.009

^aSee table IV for composition before addition.

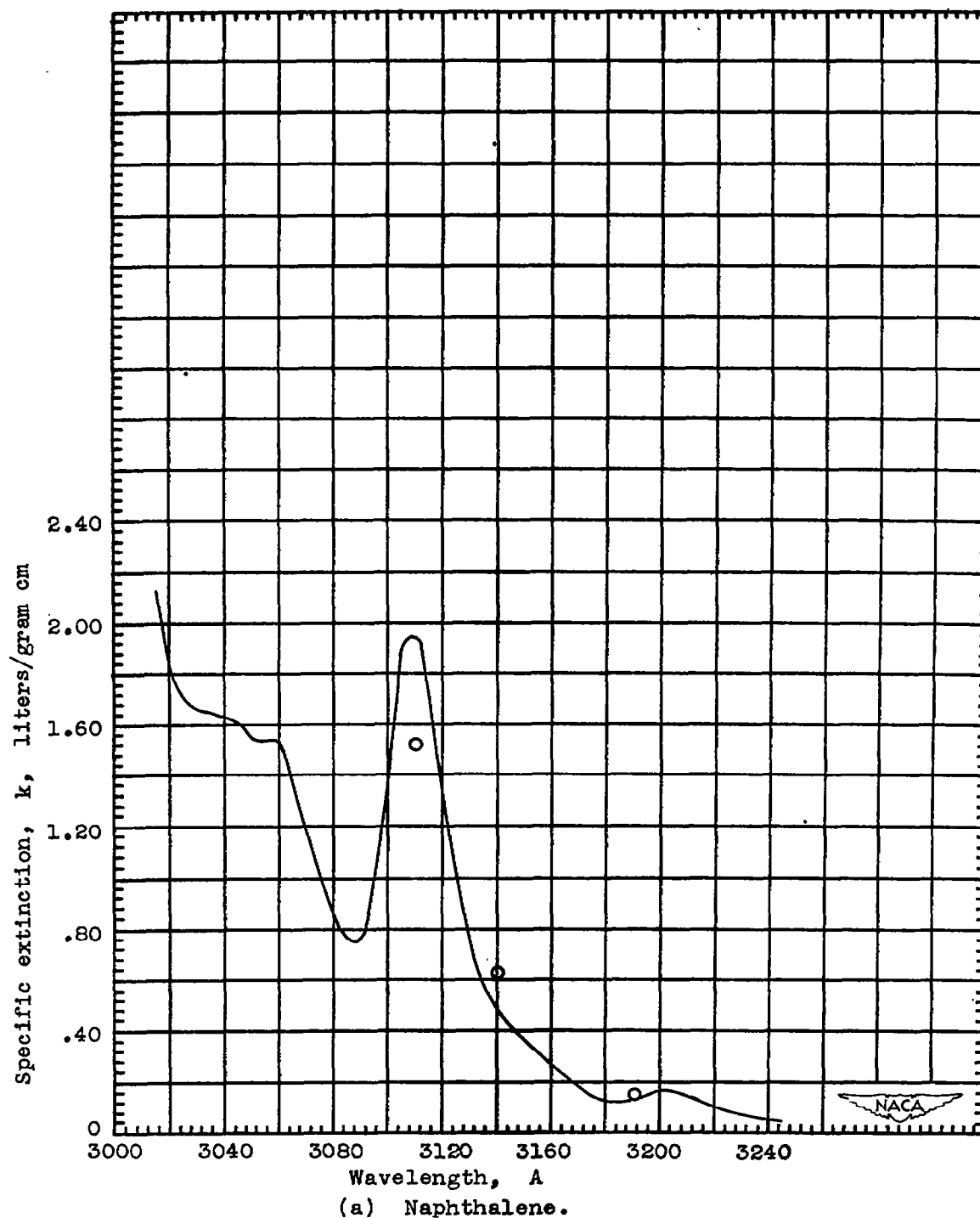
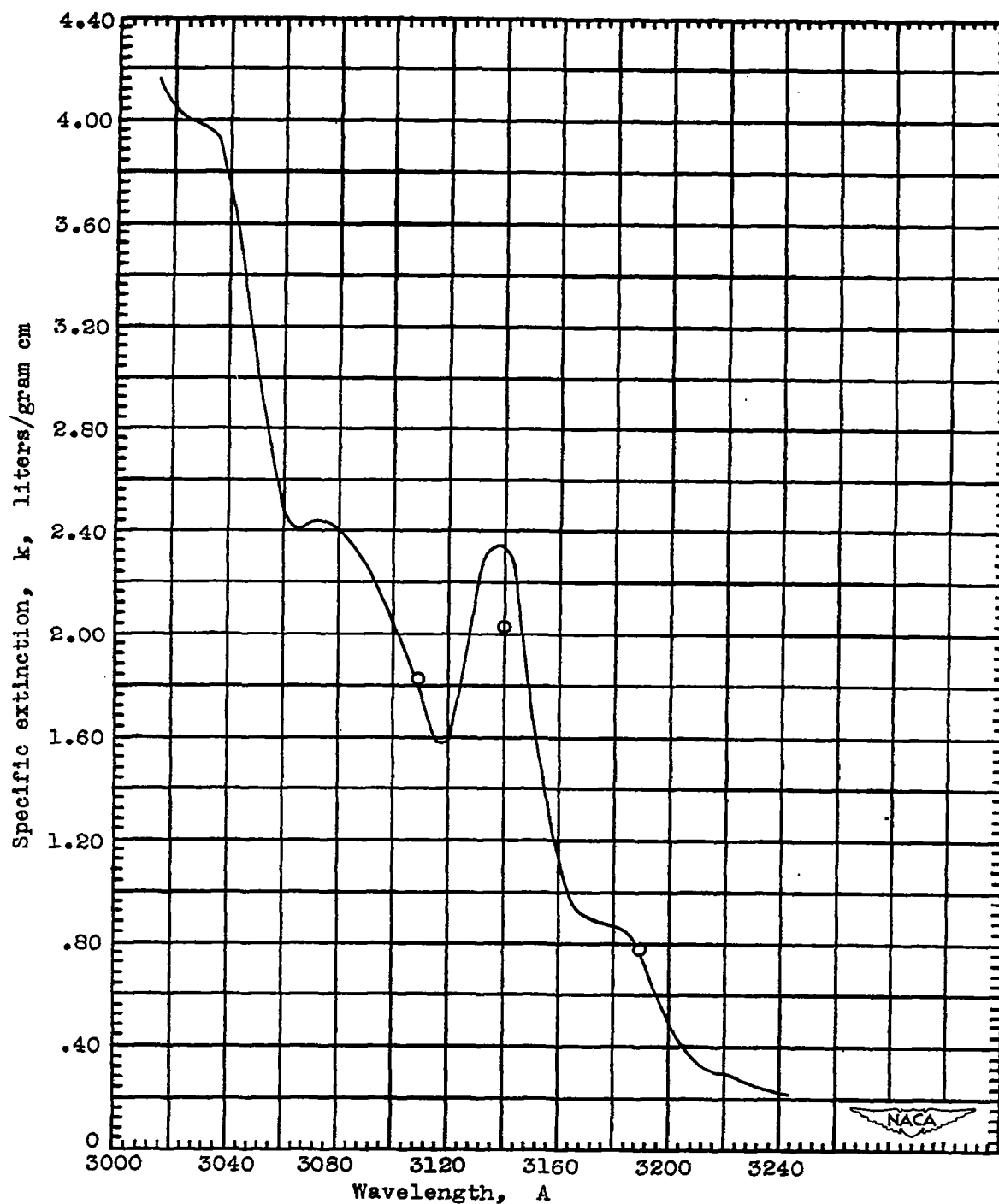
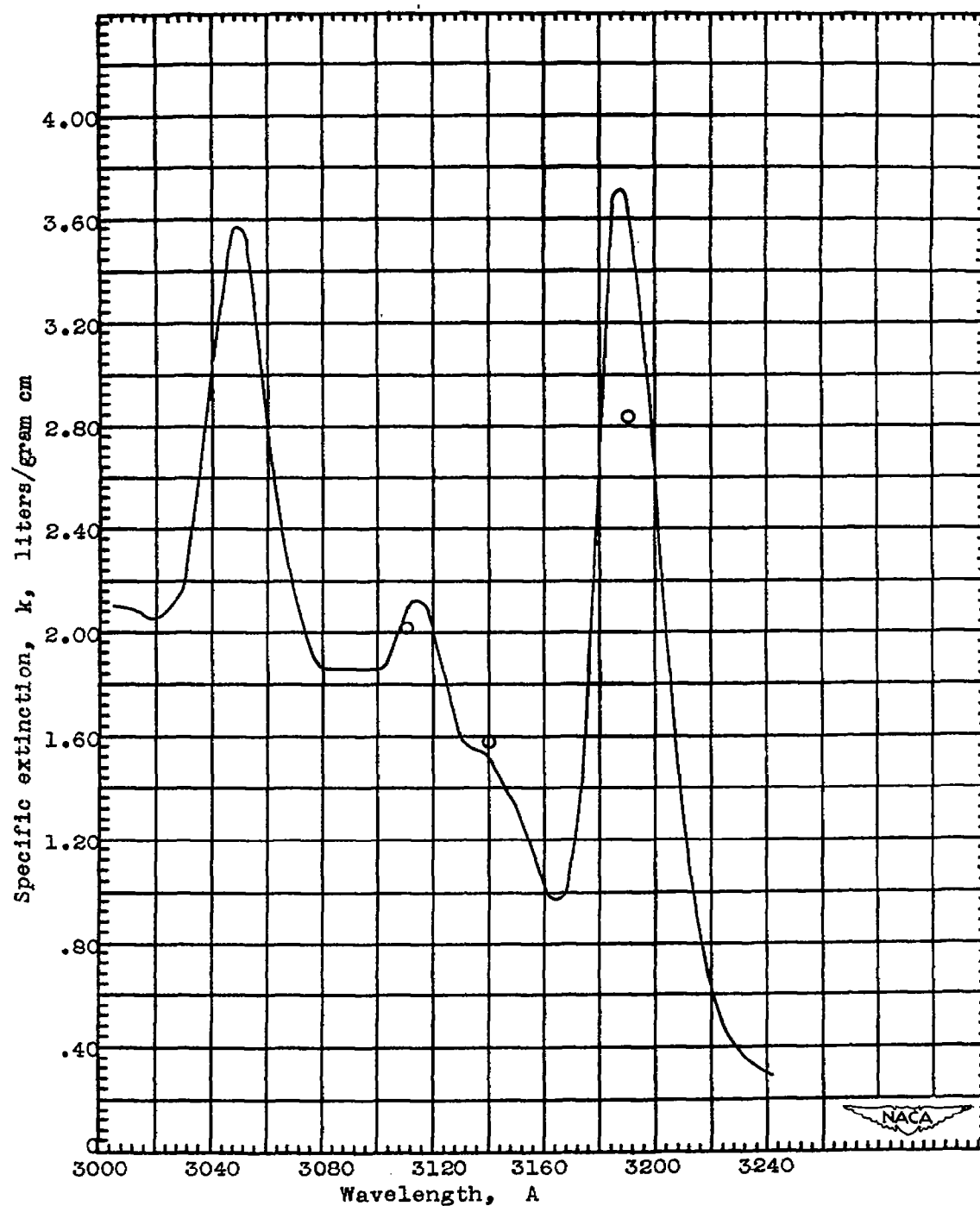


Figure 1. - Ultraviolet absorption spectra of three alkylnaphthalenes determined by photoelectric spectrophotometer. Solvent, isooctane; slit widths, 0.16 millimeter. Circles denote similar measurements with slit widths of 0.50 millimeter.



(b) 1-Methylnaphthalene.

Figure 1. - Continued. Ultraviolet absorption spectra of three alkyl-naphthalenes determined by photoelectric spectrophotometer. Solvent, isooctane; slit widths, 0.16 millimeter. Circles denote similar measurements with slit widths of 0.50 millimeter.



(c) 2-Methylnaphthalene.

Figure 1. - Concluded. Ultraviolet absorption spectra of three alkyl-naphthalenes determined by photoelectric spectrophotometer. Solvent, isooctane; slit widths, 0.16 millimeter. Circles denote similar measurements with slit widths of 0.50 millimeter.

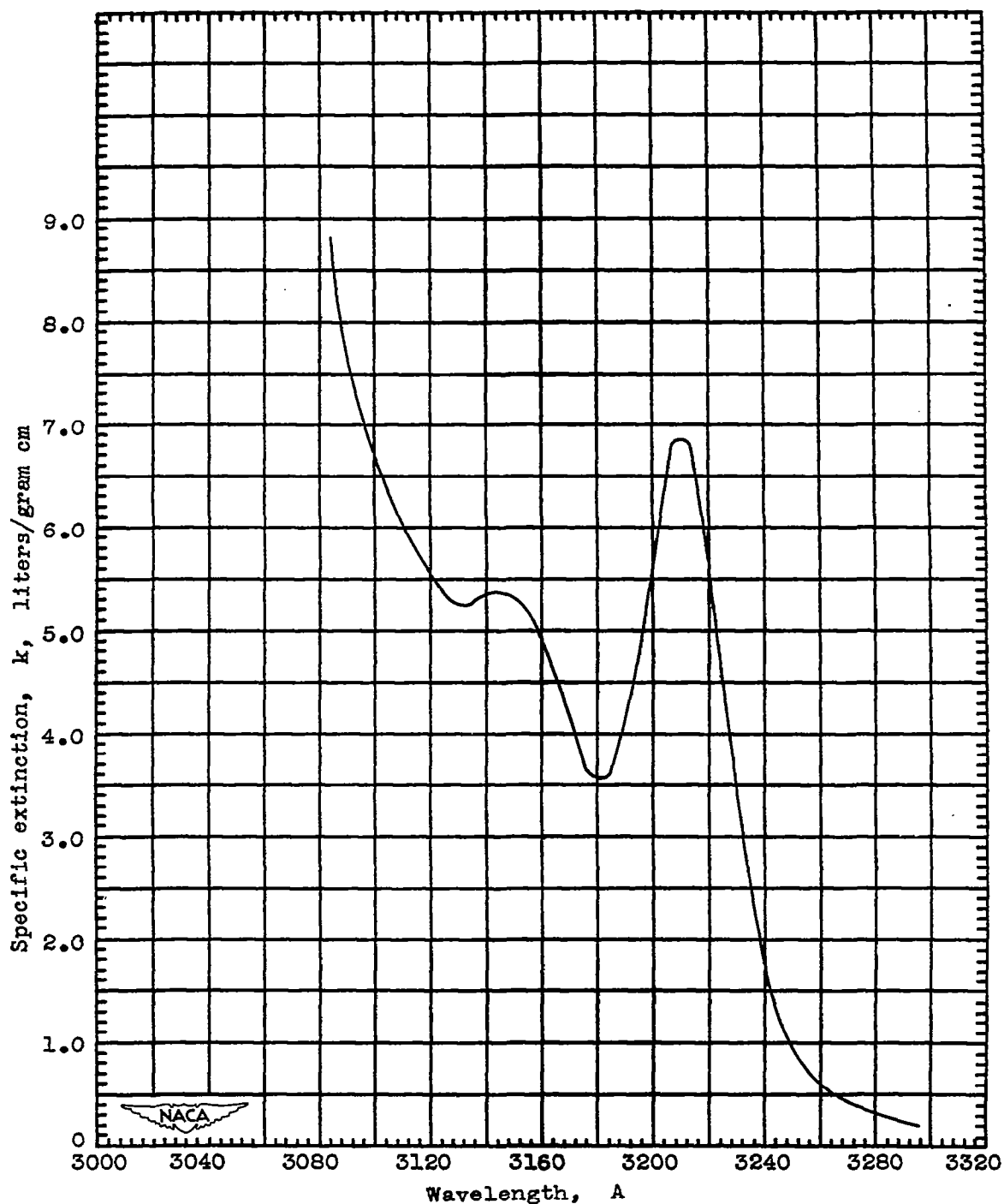


Figure 2. - Ultraviolet absorption spectrum of acenaphthene determined by photoelectric spectrophotometer. Solvent, isooctane; slit widths, 0.50 millimeter.

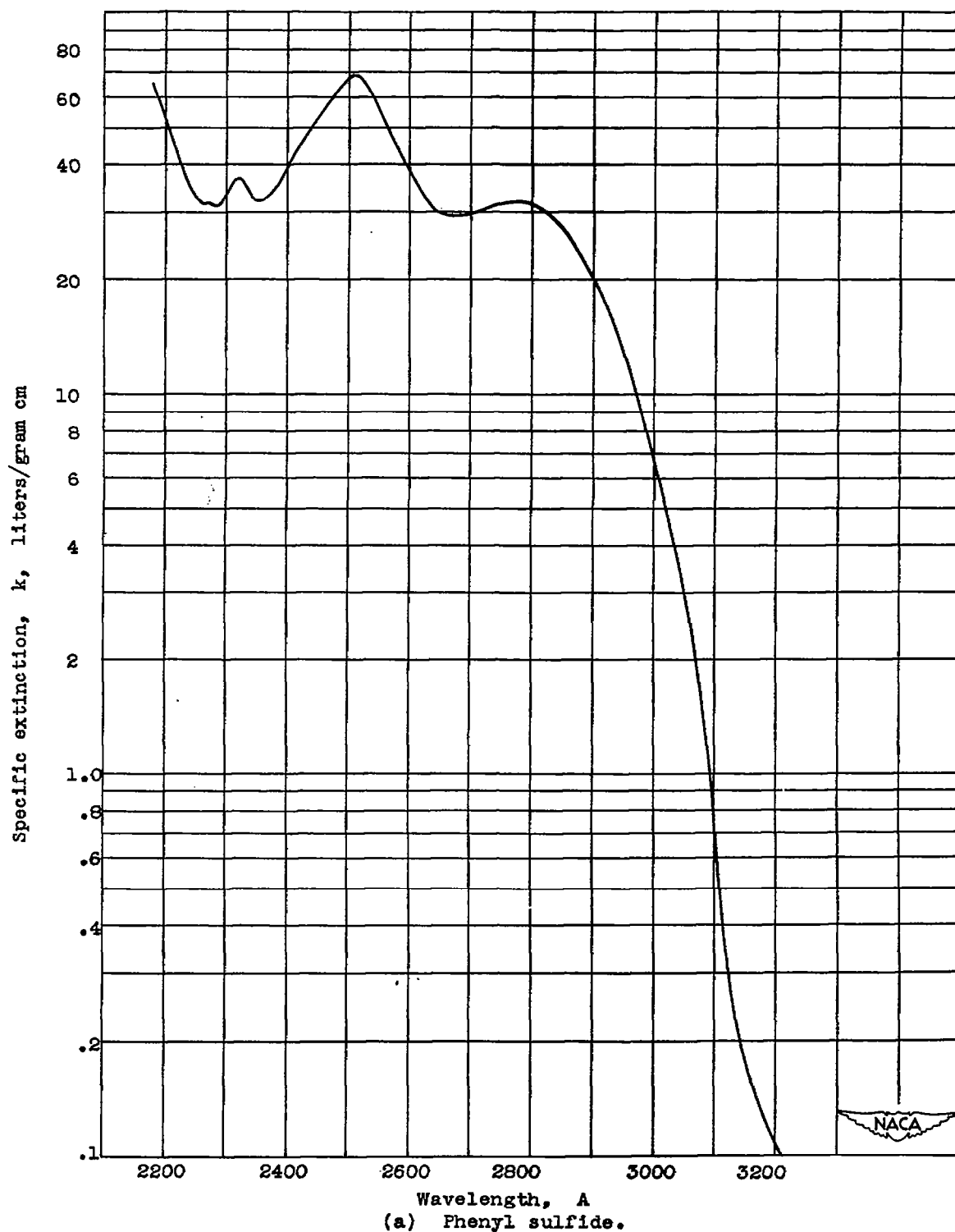
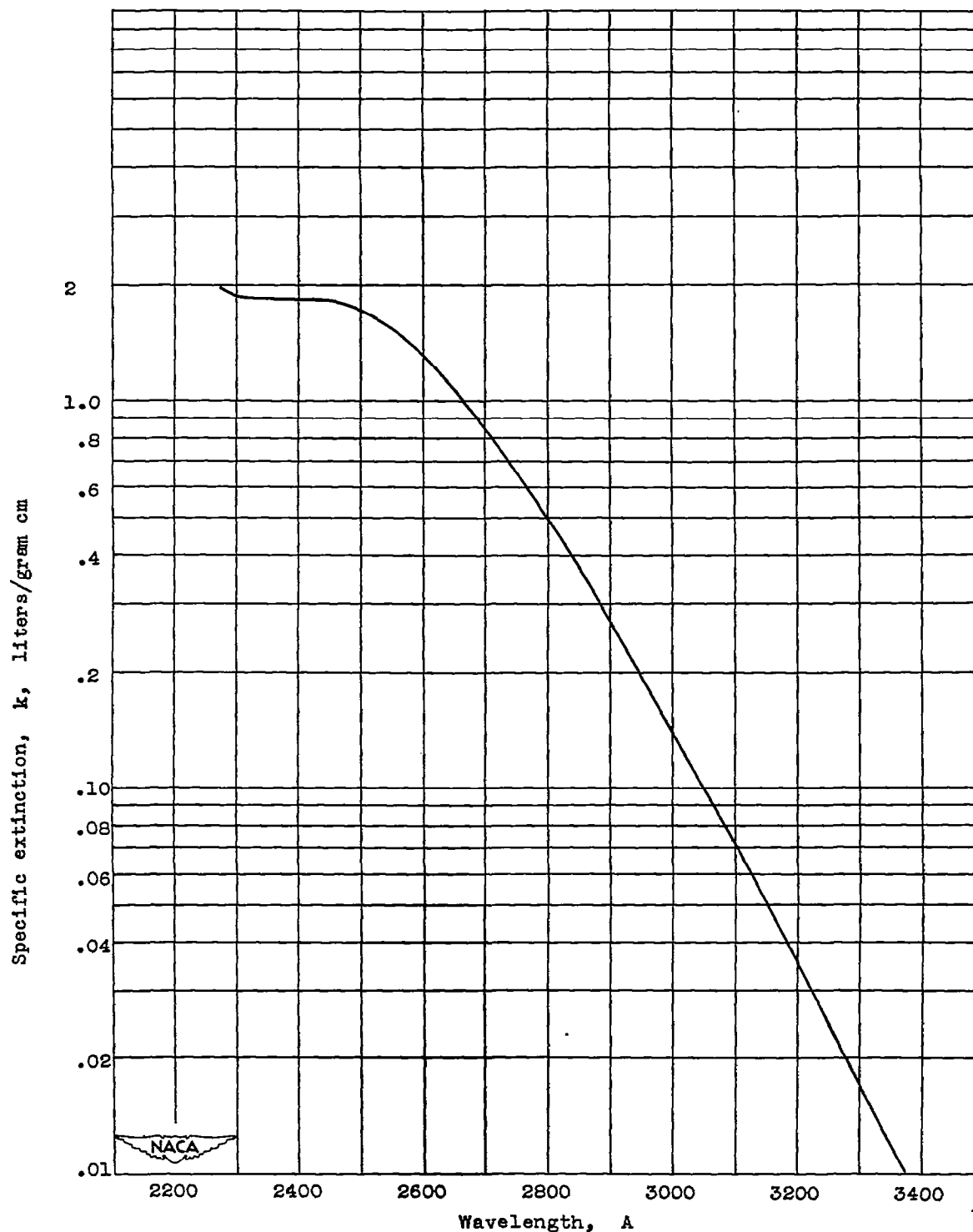


Figure 3. - Ultraviolet absorption spectra of sulfur compounds determined by photoelectric spectrophotometer. Solvent, Isooctane; slit widths, 0.50 millimeter.



(b) Amyl sulfide.

Figure 3. - Continued. Ultraviolet absorption spectra of sulfur compounds determined by photoelectric spectrophotometer. Solvent, Iso-octane; slit widths, 0.50 millimeter.

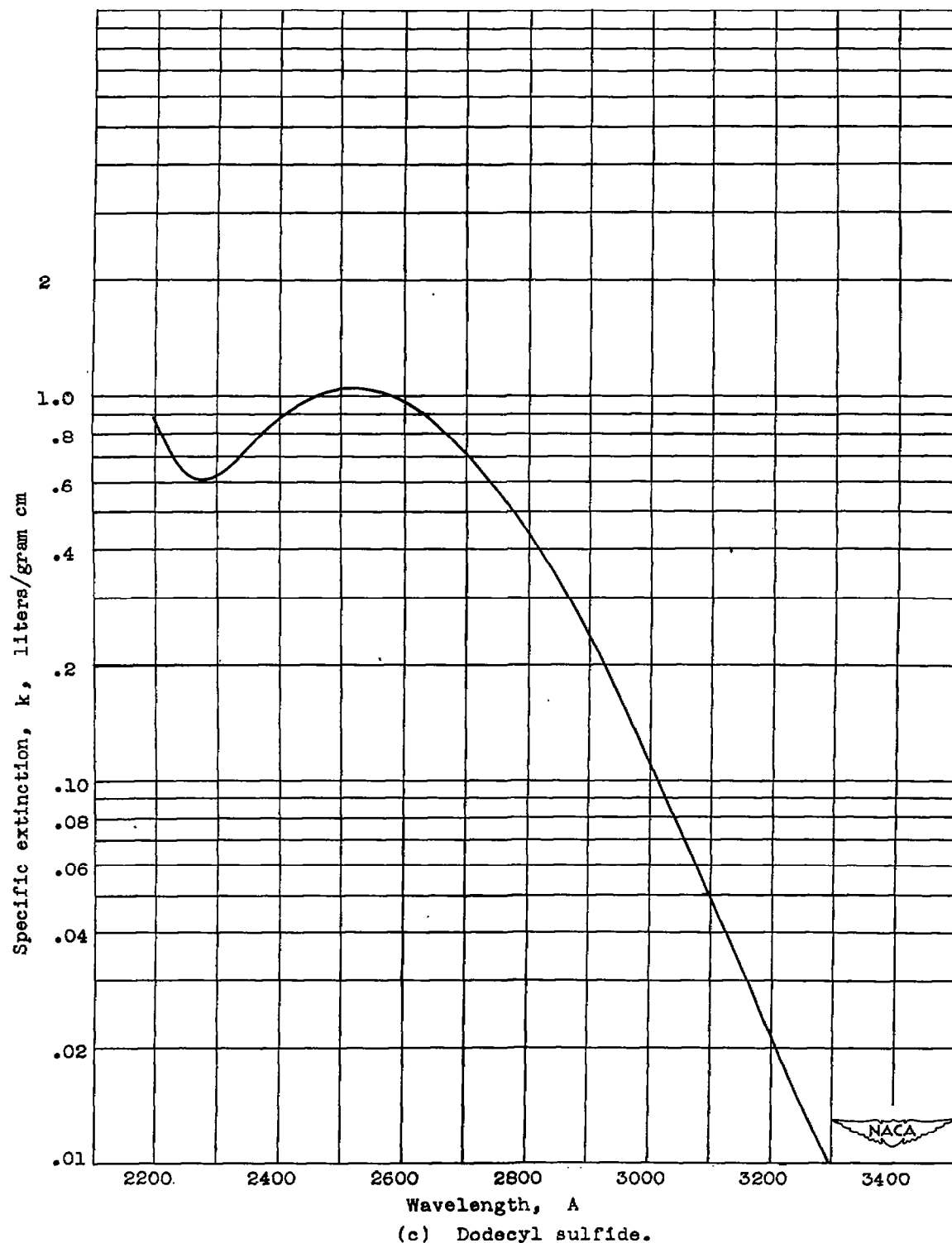
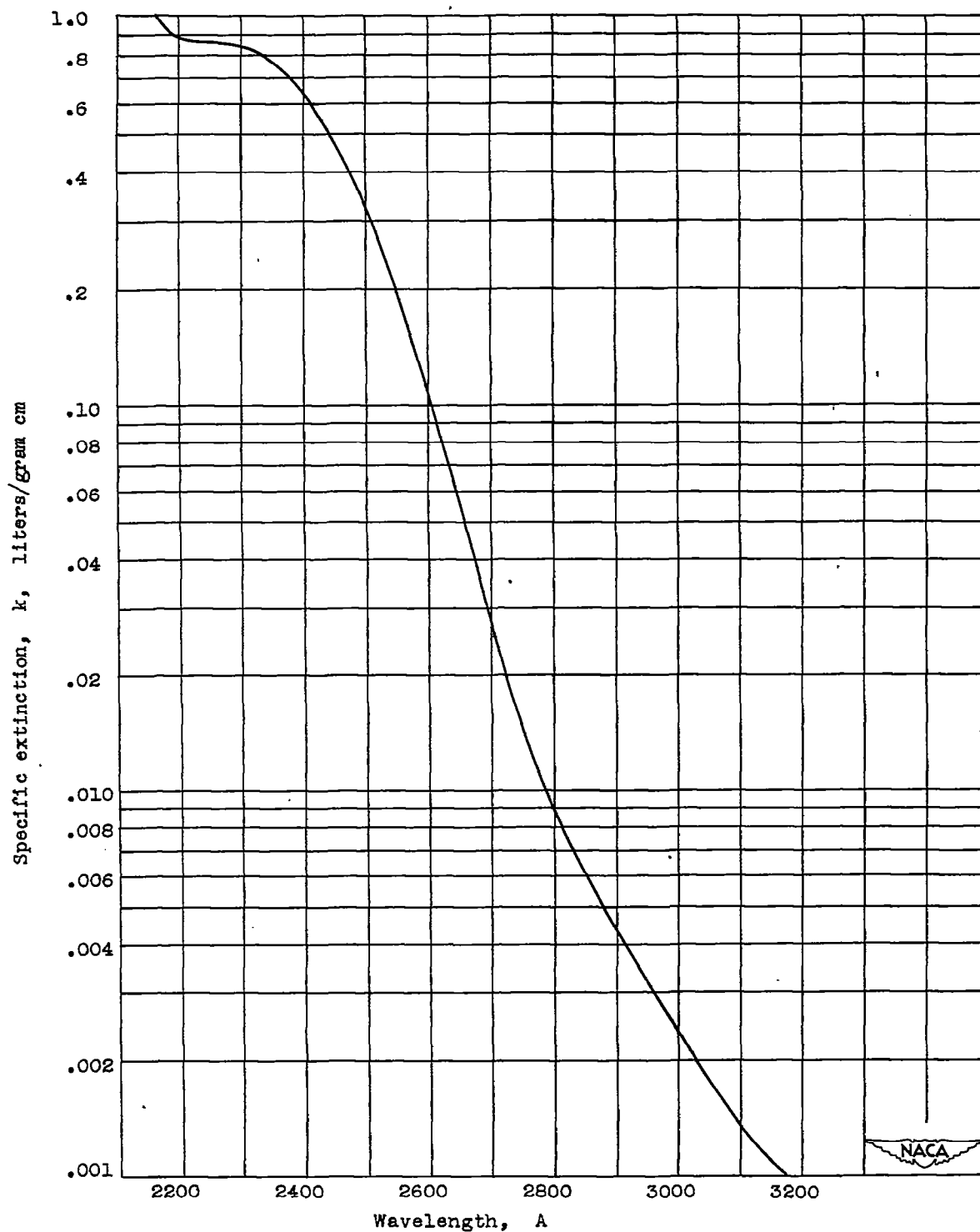


Figure 3. - Continued. Ultraviolet absorption spectra of sulfur compounds determined by photoelectric spectrophotometer. Solvent, iso-octane; slit widths, 0.50 millimeter.



(d) Dodecyl mercaptan.

Figure 3. - Concluded. Ultraviolet absorption spectra of sulfur compounds determined by photoelectric spectrophotometer. Solvent, Iso-octane; slit widths, 0.50 millimeter.

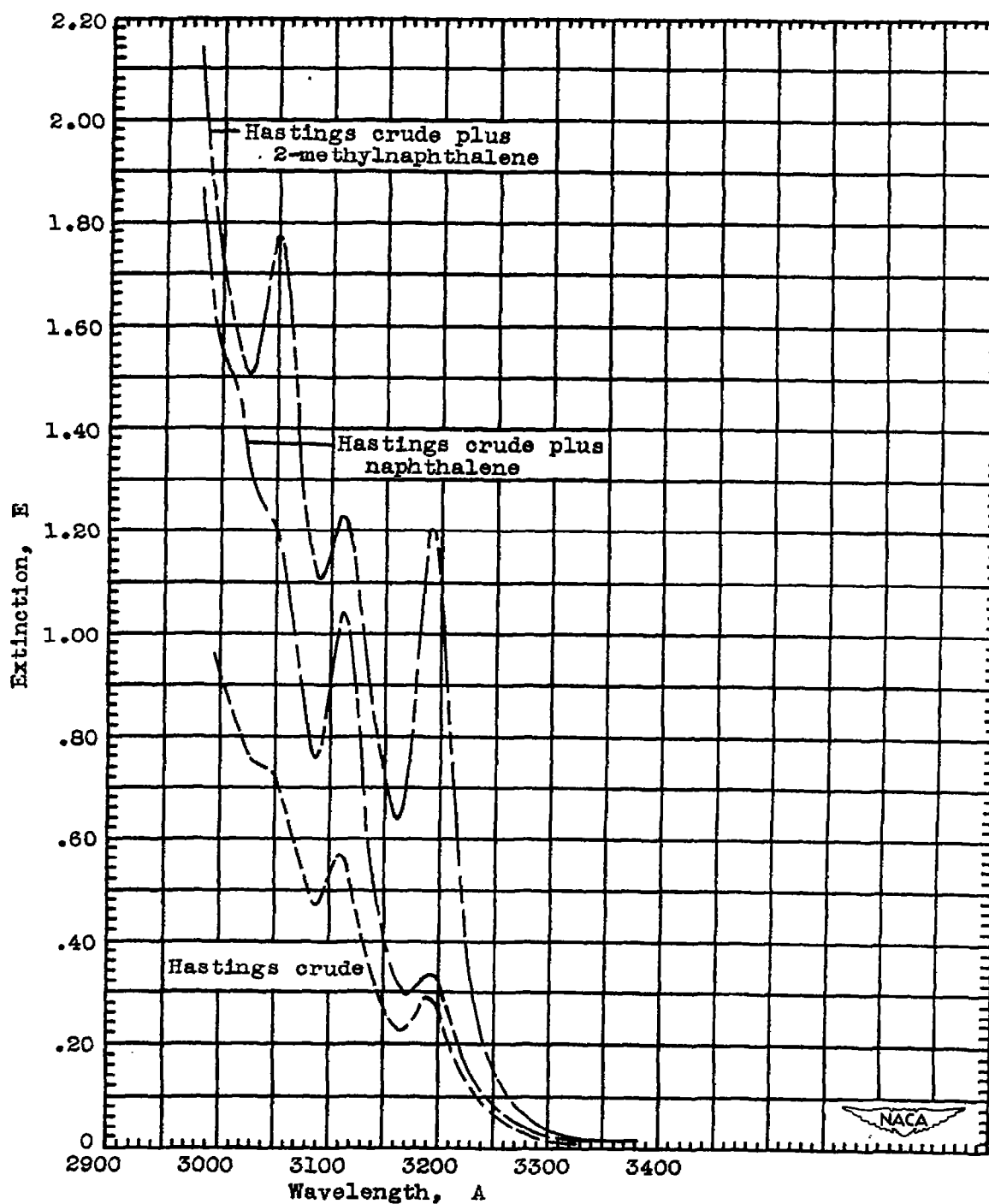


Figure 4. - Ultraviolet absorption spectrum of a 278° to 420° F cut of Hastings crude determined by ultraviolet spectrophotometer together with similar spectra resulting from addition of 0.44 percent of naphthalene by volume and 0.454 percent of 2-methylnaphthalene by volume to the cut. Solvent, isooctane; slit widths, 0.50 millimeter; cell thickness, 0.50 centimeter. Concentration of cut, 112.9 grams per liter in all three spectra.